

Sertifikaat

PATENTKANTOOR

REPUBLIC OF SOUTH AFRICA

DEPARTEMENT VAN
HANDEL EN NYWERHEID



13/10/516372
Rec'd PORTO 30 NOV 2003
Certificate
PATENT OFFICE

REPUBLIEK VAN SUID-AFRIKA

DEPARTMENT OF TRADE
AND INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

REC'D 01 JUL 2003

WIPO PCT

the documents attached hereto are true copies of the Forms P2, P6,
provisional specification and drawing of South African Patent Application
No. 2002/4382 in the names of Highveld Steel and Vanadium
Corporation Limited

Filed : 31 May 2002
Entitled : Process

**PRIORITY
DOCUMENT**

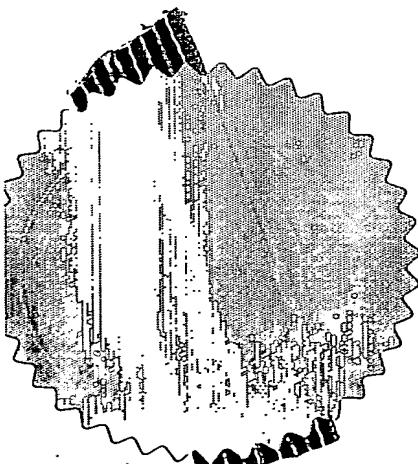
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Getekken te PRETORIA in die Republiek van Suid-Afrika, hierdie
Signed at PRETORIA in the Republic of South Africa, this

30th

dag van
day of May 2003

13/10/516372
Registrateur van Patente
Registrar of Patents
CH



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REPUBLIC OF SOUTH AFRICA

REGISTER OF PATENTS

PATENTS ACT, 1978

OFFICIAL APPLICATION			LODGING DATE: PROVISIONAL		ACCEPTANCE DATE		
21	01	2002/4382	22	31 MAY 2002	47		
INTERNATIONAL CLASSIFICATION			LODGING DATE: COMPLETE		GRANTED DATE		
51			23				
FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)							
71	HIGHVELD STEEL AND VANADIUM CORPORATION LIMITED						
APPLICANTS SUBSTITUTED:						DATE REGISTERED	
71							
ASSIGNEE(S)						DATE REGISTERED	
71							
FULL NAME(S) OF INVENTOR(S)							
72	1. DORMEHL, ANDRIES GERHARDUS 2. MONAGHAN, PATRICK ALBERT						
PRIORITY CLAIMED		COUNTRY		NUMBER		DATE	
N.B. Use International abbreviation for country (see Schedule 4)		33	NIL	31	NIL	32	NIL
TITLE OF INVENTION							
54	PROCESS						
ADDRESS OF APPLICANT(S)/PATENTEE(S)							
OLD PRETORIA MAIN ROAD, WITBANK, SOUTH AFRICA							
ADDRESS FOR SERVICE					S & F REF		
74	SPOOR & FISHER, SANDTON			PA133218/P			
PATENT OF ADDITION NO.			DATE OF ANY CHANGE				
61							
FRESH APPLICATION BASED ON			DATE OF ANY CHANGE				

APPLICATION FOR A PATENT
AND ACKNOWLEDGEMENT OF RECEIPT 05.02
(Section 30 (1) – Regulation 22)

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

HASR 505

REPUBLIC VAN SUID AFRIKA
S & F REFERENCE

OFFICIAL APPLICATION NO.

21

01

2002/4382

PA133218/P

FULL NAME(S) OF APPLICANT(S)

71

HIGHVELD STEEL AND VANADIUM CORPORATION LIMITED

ADDRESS(ES) OF APPLICANT(S)

OLD PRETORIA MAIN ROAD, WITBANK, SOUTH AFRICA

TITLE OF INVENTION

54

PROCESS

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

COUNTRY: NIL

NUMBER: NIL

DATE: NIL

THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

21 01

THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

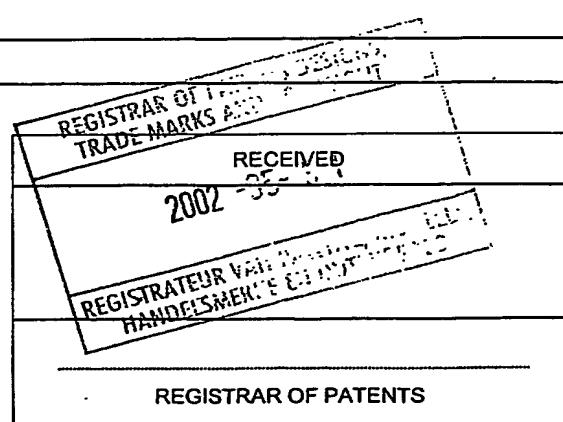
21 01

THIS APPLICATION IS ACCCOMPANIED BY:

- 1. A single copy of a provisional specification of 5 pages..
- 2. Drawings of 1 sheet.
- 3. Publication particulars and abstract (Form P.8 in duplicate).
- 4. A copy of Figure of the drawings (if any) for the abstract.
- 5. Assignment of invention.
- 6. Certified priority document.
- 7. Translation of the priority document.
- 8. Assignment of priority rights.
- 9. A copy of the Form P.2 and the specification of S.A. Patent Application No .
- 10. Declaration and power of attorney on Form P.3.
- 11. Request for ante-dating on Form P.4.
- 12. Request for classification on Form P.9.
- 13. Form P.2 in duplicate.
- 14. Other.

74 ADDRESS FOR SERVICE: SPOOR & FISHER, SANDTON

Dated: 31 May 2002


SPOOR & FISHER
PATENT ATTORNEYS FOR THE APPLICANT(S)

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

LODGING DATE

21 01 2002/4382

22 31 MAY 2002

FULL NAMES OF APPLICANTS

71 HIGHVELD STEEL AND VANADIUM CORPORATION LIMITED

FULL NAMES OF INVENTORS

72 DORMEHL, ANDRIES GERHARDUS
MONAGHAN, PATRICK ALBERT

TITLE OF INVENTION

54 PROCESS

BACKGROUND TO THE INVENTION

THIS invention relates to a process for the preparation of a vanadyl sulphate solution.

It is known to produce vanadyl sulphate by dissolving vanadium pentoxide in hot dilute sulphuric acid under vigorous agitation and continued heating with the aid of sulphur dioxide as a reducing agent.

The limited solubility of sulphur dioxide in acidic and aqueous solutions results in the emission of sulphur dioxide from the solution and this presents an environmental hazard. Overdosing of the solution with SO_2 gas results in the unwanted formation of the lower valent vanadium sulphate, namely V_2SO_4 and not vanadyl sulphate (VOSO_4).

Since the dissolution of vanadium pentoxide in sulphuric acid is endothermic heat has to be provided to drive the formation of vanadyl sulphate.

There is thus always a need for a new method for the production of vanadyl sulphate.

SUMMARY OF THE INVENTION

According to the invention a process for producing a vanadyl sulphate solution includes the steps of:

- (1) providing a starting material comprising vanadium trioxide (V_2O_3);
- (2) contacting the vanadium trioxide with an appropriate volume and concentration of a sulphuric acid solution to produce a vanadium trioxide suspension; and
- (3) contacting the vanadium trioxide suspension with a strong oxidising agent that is capable of raising the valency or oxidation state of the vanadium, thereby to dissolve the vanadium trioxide in the sulphuric acid to produce the vanadyl sulphate solution ($VOSO_4$).

Various strong oxidising agents including peroxides and permanganate such as hydrogen peroxide, sodium peroxide and potassium permanganate, for example, can be used. Hydrogen peroxide is particularly preferred as it does not introduce any impurities into the final product.

The hydrogen peroxide is typically added slowly to the vanadium trioxide suspension due to the violent nature of the reaction.

BRIEF DESCRIPTION OF THE DRAWING

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings in which:

-3-

Figure 1 is a graph indicating the mass relationship between varying quality V_2O_3 expressed as V_2O_5 against constant 4.5g V_2O_5 portions in a 4.0 molar sulphuric acid solution; and

Figure 2 indicates the reduction potential in mVolt against the mass of the V_2O_3 used.

DESCRIPTION OF A PREFERRED EMBODIMENT

The crux of the invention is to use a strong oxidising agent to dissolve vanadium trioxide (V_2O_3), commonly referred to as Hivox, in a sulphuric acid solution to produce vanadyl sulphate ($VOSO_4$).

In carrying out the process, a strong oxidising agent such as a peroxide or permanganate, for example, is used to dissolve the V_2O_3 in a warm sulphuric acid solution with constant stirring. Although various strong oxidising agents such as hydrogen peroxide, sodium peroxide, potassium permanganate, iodine, potassium iodate, potassium bromate, bromine, ammonium persulfate, persulfates of sodium and potassium, cerium (IV) sulphate, and potassium dichromate, for example, can be used, hydrogen peroxide is preferred as it does not introduce any impurities into the final product.

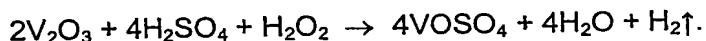
As the quality of industrial grade Hivox ranges typically from 115 to 122 percent equivalent V_2O_5 , figure 1 can be used in order to determine an appropriate quantity of V_2O_3 for use in the process, depending on the quality of the starting material used. The required solution can be obtained by monitoring the reduction potential thereof in mVolt, as shown in figure 2, during the dissolution process. The start of production of vanadyl sulphate is illustrated at the point where the graph dips sharply.

The invention will now be illustrated by way of the following non-limiting example.

Example

Hydrogen peroxide was used to dissolve 3.0 grams Hivox (V_2O_3) in a warm ($50^\circ C$) 4.0 molar sulphuric acid solution with constant stirring. The hydrogen peroxide was added dropwise as it reacted violently with the solution. During the dissolution process the reduction potential of the solution was continuously monitored and the addition of hydrogen peroxide stopped when the solution reached the end point at 600 mVolts.

The process proceeded according to the following formula:



During the process, the solution first turned green, which is indicative of the presence of V^{3+} ions, whereafter it turned blue, which is indicative of the presence of VO^{2+} ions present in the vanadyl sulphate end product.

From the above, it is evident that vanadyl sulphate can readily be made using Hivox (V_2O_3), which is generally more cost effective, and less hazardous, than the conventional process using V_2O_5 .

DATED THIS 31st DAY OF MAY 2002



SPOOR & FISHER
APPLICANT'S PATENT ATTORNEYS

2002/4382

Figure 1

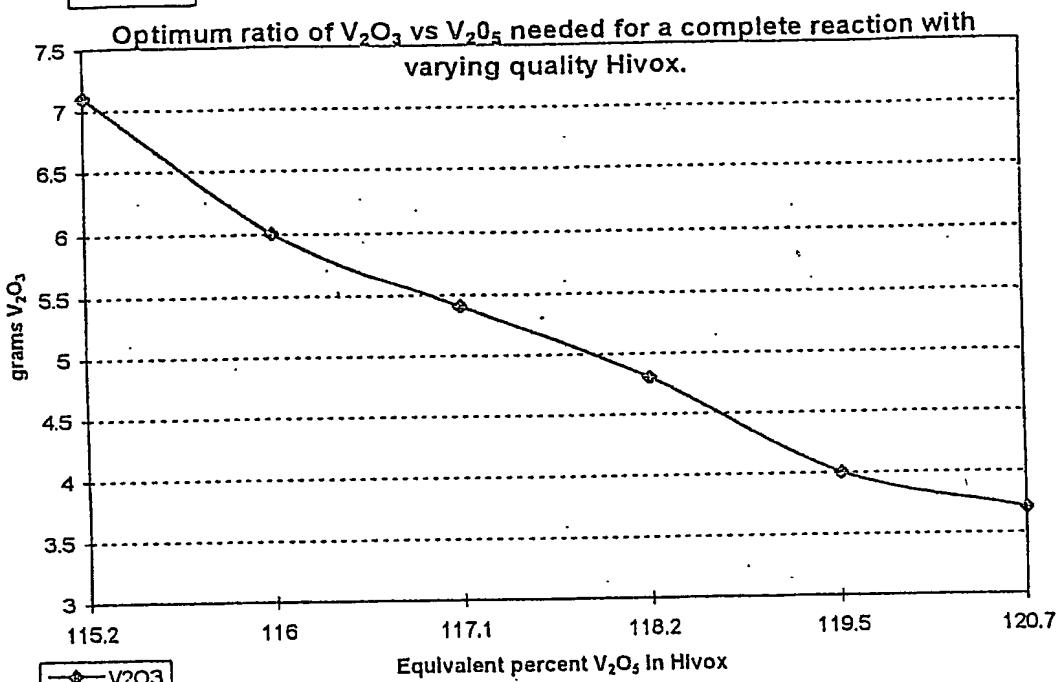
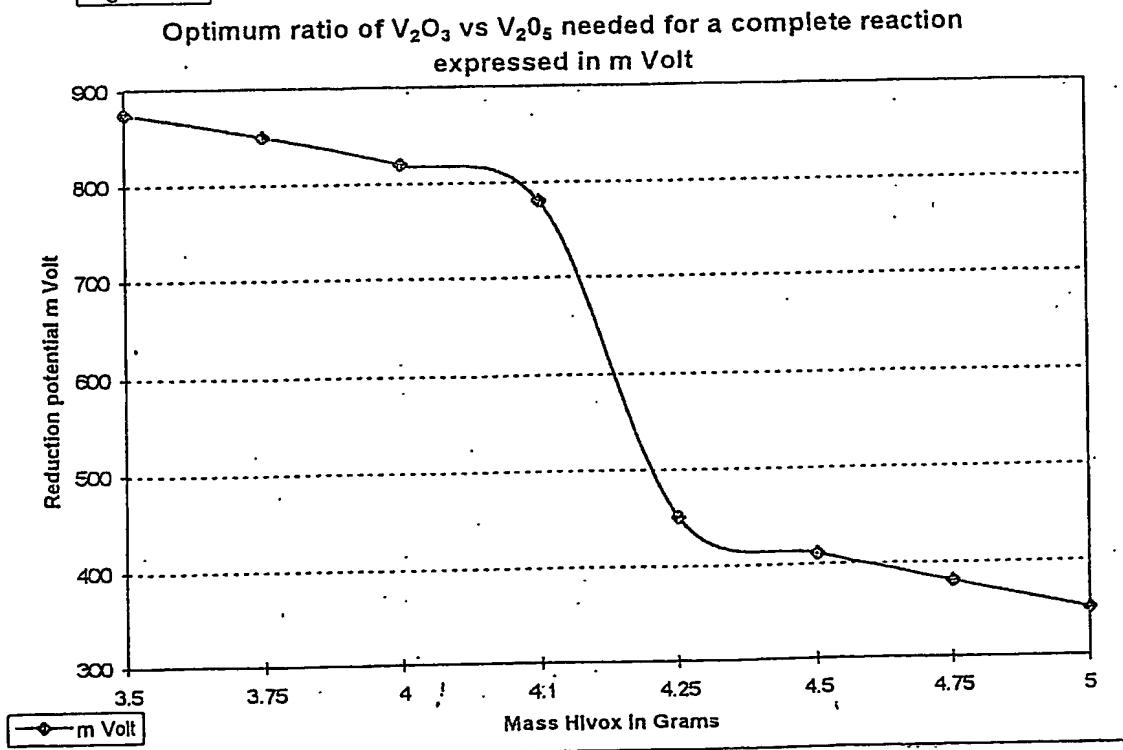


Figure 2




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Applicant's Patent Attorneys